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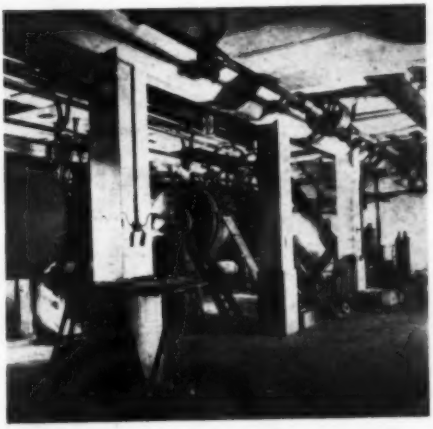

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Grading Cottonseed by Net Kernel and Fatty Acid Methods

From Addresses Delivered Before the Interstate Cottonseed Crushers' Convention and American Oil Chemists' Society

By G. S. MELOY

Last year I had the honor of discussing with you some of the arguments which had led me to advance the theory that the net kernel content of the "as is" ton of cottonseed was an index of the value of the seed. At that time, I had very little data in the way of analyses on which to prove the case. This was because in the past practically all analyses have been made on a clean seed basis. My argument, therefore, was based very largely on the results of studies of cottonseed that had been made in connection with the breeding of cotton varieties and on mathematical deductions.

I attempted to show also that if this theory of grading cottonseed were true and it were possible to construct a machine by which the kernels could be extracted from a fully representative sample, the grade could be determined at the time of loading a car as well as on its receipt at the oil mills. Check determination would therefore be possible by both shipper and receiver and the way would be prepared for the development of some plan for official sampling and grading similar to that now employed in the grading of grain.

I was limited in my discussion to the question of quantity of products possible in prime seed and did not discuss quality, since, as I stated, it was my belief that the present method of determining quality, by cutting one hundred seed and noting the percentage of kernels showing discoloration, was not only scientifically unsound but was fallacious in that it did not indicate the true quality of the oil.

During the past season, we have secured very complete analyses of over 4,000 cars of seed, representing seed produced in practically every state from North Carolina to Oklahoma. These are being carefully studied and we have already prepared several graphs from them.

Grading for Quality

First, let us consider the question of quality. Very shortly after the last convention, the American Oil Chemists' Society appointed a special committee for the purpose of studying methods of determining damage in cottonseed. This committee, as many of you doubtless already know, found that the cut method was of dubious accuracy and has brought about a new method, by which the actual percentage of free fatty acid in the oil is determined. I have been so fortunate as to secure the analyses of nearly 3,000 cars in which damage was determined by both the cutting method and by the method in which the free fatty acid in a sample of the oil is determined by titration. From these data graphs of the analyses of cars originating in each state have been made, but

I believe one or two of the graphs will suffice to illustrate the point and make it clear that the color of the kernels, which is the basis of the cutting method of determining damage, has no apparent relation to the quality of the oil.

In the first graph, the analyses of one hundred cars have been arranged in order of increasing rank of percentage of damage as determined by the cut method. These range from 100 per cent sound to 98 per cent damage, as shown by the top curve. The corresponding free fatty acid content of each of these cars is shown by the jagged line.

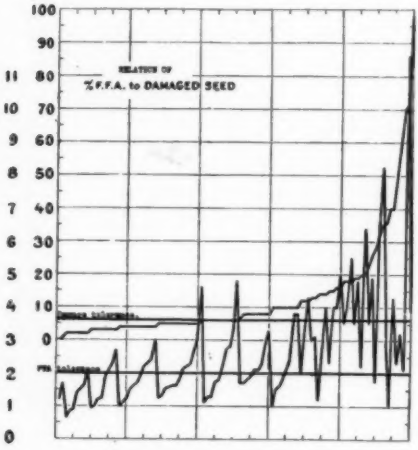

Fig. 1. Relation of damaged seed (discolored meats) to Free Fatty Acid in cottonseed oil. Analyses of 100 car lots arranged in sequence of increasing damaged seed

Out of these 100 cars, 51 were rated as prime seed by the cut method. But 18 of these 51 cars, or 35.2 per cent of them analyzed above 2 per cent free fatty acid in the oil. In two cases, the free fatty acid content was higher than was the free fatty acid in 43 of the cars that cut above 6 per cent damaged seed, including the cars that cut 40, 50, 60, 70 and 98 per cent damage. Of the 49 cars that cut above 6 per cent damage, 12 cars, or 24.5 per cent, produced oil with less than 2 per cent free fatty acid.

To sum up, in 100 cars, 49 cut off seed, or above 6 per cent damage, and practically 25 per cent of these produced prime oil. 51 of the cars cut prime seed and 35 per cent of these produced off oil.

Two of the cars cutting above 35 per cent damaged seed produced prime oil. 23 of the cars cut 15 per cent or more damaged seed but only 8 of these showed above 4 per cent free fatty acid. Of the 56 cars of off oil 19 cut prime seed. Of the 49 cars of damaged seed, 12 gave prime oil. The car cutting the highest damaged seed showed less than 4 per cent free fatty acid and nearly 1 per cent less free fatty acid than 2 cars that cut prime seed.

In the second graph, the opposite picture has been drawn. Here are shown the analyses of 139 cars, all of which tested 2 per cent or more of free fatty acid. These analyses have been arranged in rank of increasing free fatty acid. The corresponding percentage of damage as found by the cut method is shown in the irregular line. Of these 139 cars, we find that 54 cars, or 38.8 per cent, cut prime and yet 45 of them contained above the tolerance of free fatty acid.

These two graphs are sufficient to show that the cut method of determining damage has no scientific basis and its use more frequently inures to the damage of the oil miller than to the ginner or producer of seed. The method has no apparent place at present in evaluating cottonseed. Possibly it may be found to have a place in evaluat-

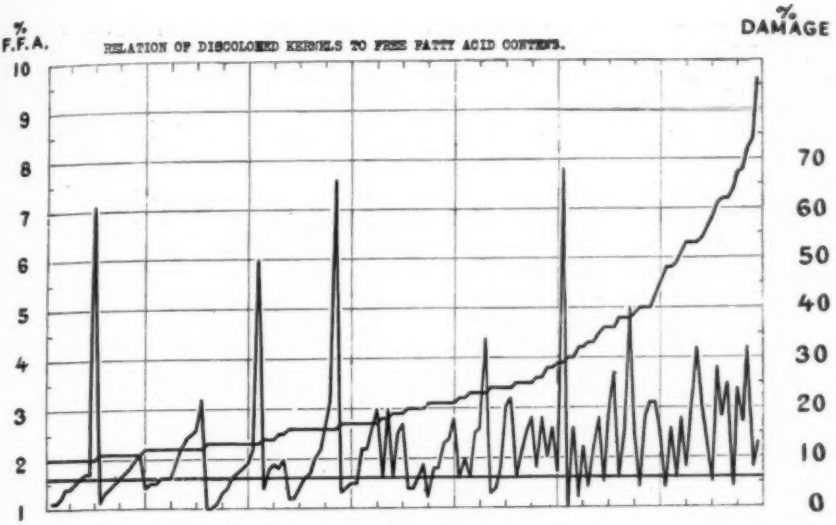

Fig. 2. Relation of Free Fatty Acid in cottonseed oil to discolored kernels (damaged seed). Arranged in sequence of increasing Free Fatty Acid

ing the color and other qualities of the meal. The direct determination of the free fatty acid content seems to be a much more sound method and has, moreover, the advantage, as your chemists will advise you, of having a direct relation to the color and manufacturing loss in the oil produced.

So much for the question of quality. Let us turn to the question of quantity or the quantitative grading of cottonseed.

Quantitative Grading

It has been impressed on me on several occasions that possibly the greatest obstacle to an understanding of the theory of grading cottonseed on the basis of their net kernel content is the difficulty of getting those engaged in the industry to think in terms of anything other than averages. Last fall I sent out a number of letters asking that certain specific data, relative to receipts of seed be kept during the current season, so that I might have material for study. In reply, generally I was referred to reports of *averages*, but I wanted data from which not only the averages for the season would be calculated, but new and unusual analyses for the purposes of these studies.

According to the dictionary, an average is "a mean proportion made out of unequal sums or quantities."

What are the unequal quantities, related to raw cottonseed, that should be considered in making the averages of seed purchases? In cottonseed we find inequalities in three characters: First, there is an inequality in the percentage of oil in the meats. Second, there is an inequality in the percentage of protein in the meats. And, third, there is an inequality in the percentage of meats in the seed.

Now, next let us see if there are any relationships between these inequalities. We do find a general and fairly constant relationship between the percentage of oil and

the percentage of protein. The relationship is in inverse ratio. That is, the higher the percentage of oil, the lower the percentage of protein, and vice versa, the lower the percentage of oil the higher the percentage of protein.

But there appears to be no relation between either the percentage of oil, or of protein, in the meats and the percentage of meats in the seed. Therefore, we may have different lots of seed contrasted as follows:

Seed with:

(A) High meat percentage and high-oil-low-protein.

(G) Seed falling between each of these.

Regardless of the current value of the oil and meal, the greatest difference in the values of any two lots of seed will be found between that lot which analyzes the extreme low kernel and oil content (F) and that other lot of seed which analyzes the extreme high kernel and oil content (A).

Suppose we call this extreme difference in value a "100 per cent spread."

Now let us compare the values of the products obtainable from two lots of seed analyzing as "A"

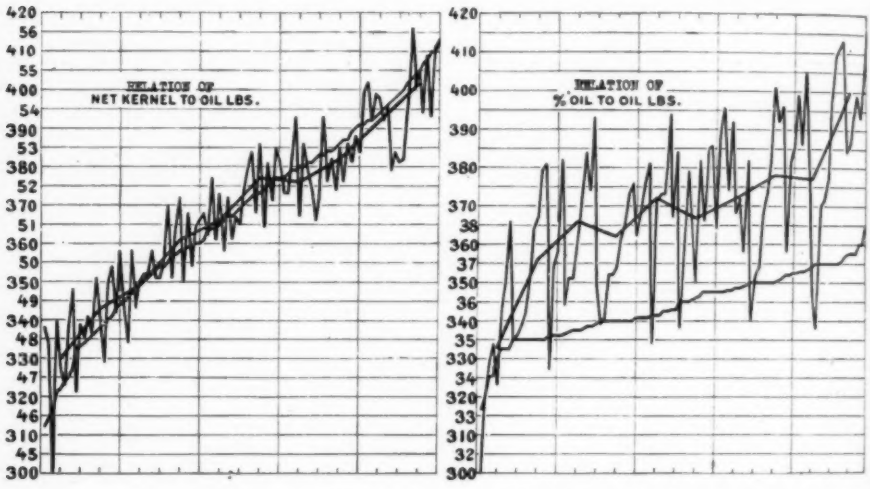

Fig. 3. Relation of the net kernel percentage of "as is" cottonseed to the total available pounds of oil. Right, same car lots arranged to show the relation of the percentage of oil in the meats to the total pounds of oil

(B) High meat percentage and low-oil-high-protein.

(C) Average meat percentage and high-oil-low-protein.

(D) Average meat percentage and low-oil-high-protein.

(E) Low meat percentage and high-oil-low-protein.

(F) Low meat percentage and low-oil-high-protein.

and as "B". The difference in value is approximately 24 per cent of this total possible spread.

With two lots analyzing as "C" and "D", the difference in value of the products is approximately 23 per cent of the total possible spread.

With two lots analyzing as "E" and "F", the difference in value is approximately 18 per cent.

But when we compare a lot of seed analyzing as "A" with another lot analyzing as "E", we find the difference in value of the products is approximately 82 per cent of the total possible spread.

A comparison of seed analyzing as "B" with those analyzing as "F" shows an approximate difference in value of 77 per cent of the maximum spread.

These differences are based on the extremes of oil-in-meats percentage (25 per cent to 39 per cent) and the average range of meat content. A comparison based on the usual differences in the oil-in-meats percentage makes the differences in values of the products even more contrasted. In the East during the last season about 90 per cent of the analyses showed between 34 per cent to 37 per cent of oil. In the West this runs somewhat lower, approximately 30 per cent to 33 per cent.

Possibly it might make this argument a little easier to understand if we assigned definite values in place of percentage of spread. With oil at 10 cents and 7 per cent meal at 2 cents the difference in value of the oil and meal between two tons of seed, one analyzing 42 per cent net kernel and 34 per cent oil and the other analyzing 59 per cent net kernel and 37 per cent oil, is approximately $20.00. Call this $20.00 our maximum spread.

Then the difference in value between two tons of seed analyzing as "A" and "B" would be $1.56. Between two tons analyzing as "C" and "D" $1.26, and between two tons analyzing as "E" and "F" $1.05. But the difference in value between two tons analyzing as "A" and "C" is $10.00 and between two tons analyzing as "B" and "D" is $9.70, and between "C" and "E" seed $8.95, and finally, between two tons analyzing as "A" and "E" the difference in value of the oil and meal is $18.94.

The outstanding conclusion to be reached from these figures is that in purchasing cottonseed for crushing, the kernel or meats content is the principal factor or value, since the differentials on account of oil percentages are nominal in comparison with value differentials on account of kernel percentages.

Let me remind you that for the purpose of grading seed on the net kernel content, I am proposing to consider everything as foreign matter that is not kernel, or that has to be specially processed in order to secure the kernel, such as grabots. Therefore, in preparing the graphs from the analyses secured this season, I have added together the percentage of excess moisture, the percentage of dirt and trash and the percentage of grabots and thus found the number of pounds of foreign matter in the seed. But this leaves another form of foreign matter; immature or aborted seed. Immature and aborted seed are stated in percentages, but, as you know, it is not a percentage of weight but a percentage of number. After a number of trials, I found that immature seed weighed approximately 0.36 per cent of the weight of sound seed, or 7.2 pounds per ton for each numerical percentage; therefore, to the total pounds of foreign matter found as above, 7.2 pounds was added for each per cent of immature seed. From this the net pounds of seed was ascertained, then, using the percentage of kernels in clean seed, the net pounds and percentage of kernels in the "as is" ton were easily calculated.

Having determined the net kernel content and knowing the percentage of oil in the meats, the total

pounds of oil in the "as is" ton was readily calculated.

In this way similar data was calculated on several hundred cars of seed originating in each state. For the purpose of graphs, this number was reduced to 100 analyses, by throwing out the duplicates and the near duplicates. Then the analyses were arranged first in order of increasing rank of net kernel percentage with the corresponding pounds in oil, as shown on the left side of Graph No. 3. You will note the close relation of the pounds of oil to the net kernel. Next, the analyses of the same cars were arranged in the rank of increasing percentage of oil in the meats with the corresponding total pounds of oil, as shown on the right side of the graph. In the first case, where the analyses are arranged in order of increasing percentages of kernel, the variation in the pounds of oil is due to the differences in the percentages of oil. Notice the narrow range of variation as compared with the variation in the pounds of oil when arranged in rank of increasing oil percentage, where the variation is due to the differences in the net percentages of kernels.

In one case I went a step further and calculated the total value of the oil and protein or cake as shown in Graph No. 4, in which I have arranged the analyses of the same 100 cars of seed in four different ways. First, in rank of increasing percentage of oil in the meats with the corresponding values of oil and meal. Second, in rank of increasing net kernel content with the cor-

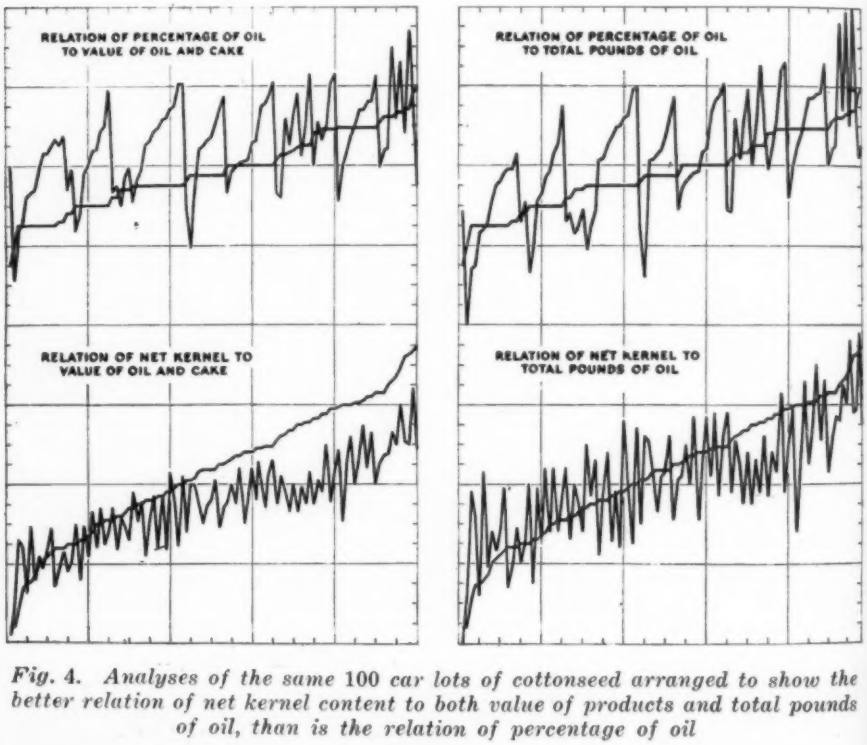
RELATION OF PERCENTAGE OF OIL TO VALUE OF OIL AND CAKE
RELATION OF PERCENTAGE OF OIL TO TOTAL POUNDS OF OIL
RELATION OF NET KERNEL TO VALUE OF OIL AND CAKE
RELATION OF NET KERNEL TO TOTAL POUNDS OF OIL

Fig. 4. Analyses of the same 100 car lots of cottonseed arranged to show the better relation of net kernel content to both value of products and total pounds of oil, than is the relation of percentage of oil

responding values of the oil and meal. Third, in rank of increasing percentages of oil with the corresponding total pounds of oil, and fourth, in rank of increasing net kernel content and the corresponding total pounds of oil.

Where the graphs are arranged on increasing oil percentages, the variations in value are due to differences in net kernel content and to variation in protein. The variations in total oil are the result of differences in the net kernel. Where the graphs are arranged in increasing net kernel content, the variations in value are the results of differences in the oil and protein content and in total pounds of oil resulting from differences in the percentages of oil.

You will notice that the variations in value of the products as compared to the pounds of oil are more narrow. This is because of the variable protein factor.

I believe that these charts will bear me out that the net kernel content of the "as is" seed is more frequently and a closer index of value of cottonseed than any other single factor. And it is logically so, since it is a direct index of the amount of both the oil and the cake, the two principal and frequently co-ordinate products of your industry. Moreover, the net percentage of kernels when used for grading purposes has very distinct advantages, as pointed out in my address at Asheville on July 1, last, in that it carries premiums for good cultural and ginning practices as well as penalties for the opposite, poor cultural practices and indifferent and culpable practices at ginneries.

The graphs I have used are from seed originating in South Carolina. I have graphs for seed originating in some of the other states which I will be glad to show all who may be interested.

Practical Application

This brings us to the practical application of the theory of grading in the purchasing of cottonseed. First, we should decide upon a base. The average kernel content of clean seed for all analyses I received this year was 54 per cent. Suppose we split the difference between a theoretical average of 50 per cent and a practical average for clean seed of 54 per cent and make our base 52 per cent net kernel in the "as is" ton. If 52 per cent net kernel had been established as the basis during last season, the cars for which I received analyses would have fallen into the following groups:

31 per cent would have received premiums.

19 per cent would have received the basis price.

50 per cent would have received discounts.

The premiums would have been paid for clean seed of better than the average quality, and the discounts would have been based on the inferiority of the shipments resulting from the growth of nondescript varieties and excesses of foreign matter.

You will recall that last year in my discussion of the scheme of grading, I stated that the basis grade and the grade limits could be determined only after sufficient actual analyses had been made in practice. Two changes are now being proposed on the basis of the analyses we now have: First, that the basis grade be 52 per cent net kernel instead of 50 per cent, and second, that each percentage of increase and decrease in the net kernel carry its appropriate premiums and discounts. At first, I suggest-

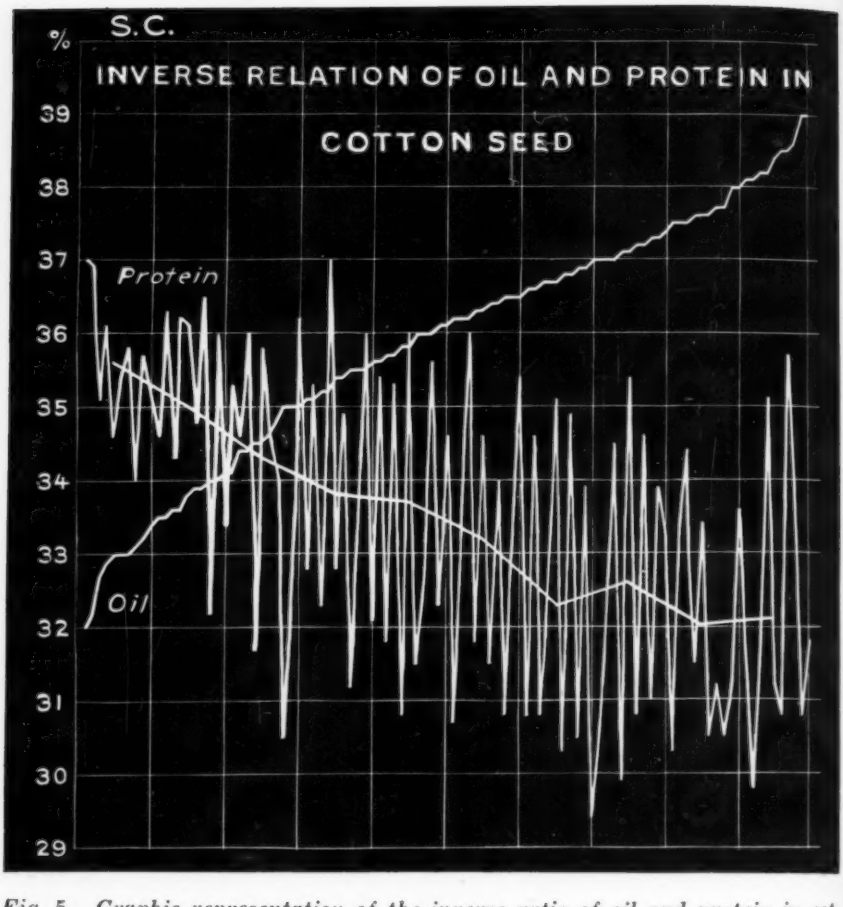

Fig. 5. Graphic representation of the inverse ratio of oil and protein in cottonseed as shown by analyses of seed from South Carolina

ed that each grade comprehend three per cent of net kernel, but experience has shown that the value differentials would be too wide and I am accordingly now suggesting single percentages of net kernel as the basis of the grades.

There is one other chart in which you may be interested, that shown in Fig. 5, above. This shows in graphic form the inverse relationship between oil and protein in cottonseed. This is not offered as a new discovery but I believe it has not been brought to your attention in graphic form before this. The results of analyses for oil are plotted on the ascending curve and the progressing average of protein analyses is represented by the line descending from left to right.

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The Behavior of Soaps of Various Oils on Dilution

Castor Oil Soaps Exhibit Marketd Resistance to Hydrolysis with Water

By H. B. STOCKS

IN a paper read before the Society of Public Analysts, London, and published in the "Analyst" December, 1923, page 590, the author proposed a new test for castor oil, and the detection of any other oil which might have been added for the purpose of adulteration. This test is based on the observations made when soaps were diluted with water.

It is a well known fact that when even neutral soaps are dissolved in water, hydrolysis takes place and free alkali is formed. This was first discovered by Chevreul and has been confirmed by numerous observers. Chevreul found that when neutral sodium and potassium salts of stearic and palmitic acids were dissolved in large volumes of hot water, on cooling crystals separated having the composition of acid salts, i.e., half the sodium or potassium in the salts was replaced by the equivalent of hydrogen, the salts being represented by the formulæ $Na.H.R_2$.

Soap Solutions Unstable

The products of the ionization of ordinary salts in solution, for instance NaCl, Na_2SO_4, etc., do not give a pink color with phenol-phthalein. In both these cases a strong base is combined with a strong acid. The same may be said of the salts of many organic acids, including the lower members of the saturated fatty acid series—acetic, butyric, etc.—which, in solution in water, can be titrated to neutrality and estimated with phenol-phthalein as indicator. But with the soaps the problem is an entirely different one. The higher fatty acids are insoluble in water, and although they form neutral salts or soaps with alkalies, these are hydrolyzed more or less according to the amount of dilution, i.e., they separate into two phases, one of which is acid and the other correspondingly alkaline. A hot solution may be, and generally is, quite clear, but addition of phenol-phthalein shows that free alkali is present, the acid soap being too weak to exert the opposite effect on the indicator to any marked extent. On cooling the solutions usually become turbid and a suspension of the acid phase in an alkaline medium is the result. In the case of pure acids of high melting point, i.e., palmitic, stearic, etc., the acid phase separates in the form of fine crystals having a satiny lustre which remain suspended in the liquid, but which can readily be separated by filtration, the clear filtrate being strongly alkaline, containing half the total alkali, and practically free from soap.

With ordinary commercial soaps this separation is not so clearly marked, and it is impossible to separate, except incompletely, the two phases, but there is reason to believe that in these cases the change is substantially the same.

The pink color which develops on addition of phenol-phthalein to solutions of most soaps deepens as

dilution is increased, but the writer found that with castor oil soaps only a faint pink color was produced which did not deepen appreciably on dilution. On titrating these former solutions with standard acid it was found that the hydrolysis alkali increased up to a maximum at a certain dilution, (i.e. part of soap in about 150 parts of water), further dilution causing little or no change. The titrations are not entirely perfect since a faint pink color usually reappears showing that equilibrium is only momentary, but on increasing the dilution beyond that above mentioned the end point is more distinct, a faint pink color reappearing in some cases only after an appreciable interval of time. With most of the soaps examined by the writer the hydrolysis alkali amounted approximately to 50% of the total, but castor oil soap formed a marked exception, the hydrolysis alkali in this case amounting to not more than 5% of the total.

The following results, given in the original paper, will illustrate the behavior of various soaps with this test:—1 gram of soap dissolved in water and made up to 320 cc. for titration.

Soap	Free Na_2O found A	Total Na_2O found B	Per cent of total A × 100 / B
Stearic acid	0.056	0.111	50.5
Palmitic acid	0.057	0.119	48.0 (1 part in 160)
Oleic acid	0.049	0.103	47.5
Yellow bar soap	0.037	0.082	45.1
Olive oil soap	0.043	0.083	52.3
Castor oil fatty acids	0.004	0.093	4.3

The first three were made by dissolving the pure acids in the calculated quantities of sodium hydroxide solution, the first two contained slightly more than 1 gram of soap, but this in no way affects the experiments.

More recently the soaps of several other fatty acids have been examined, but, in order to simplify matters, instead of 1 gram of the salt or soap, 1 gram of the fatty acid was taken, dissolved in alcohol, neutralized with sodium hydroxide solution to phenol-phthalein and the liquid evaporated to dryness. The salt or soap was dissolved in boiled distilled water, diluted to 350 cc. and then titrated with N/10 hydrochloric acid until neutral to phenolphthalein, then in presence of methyl orange to complete neutrality.

The following results were obtained:

Salt of soap	Free Na_2O found A	Total Na_2O found B	Per cent of total
Acetic X	0.00329	0.5128	0.64
Butyric X	0.00282	0.3444	0.82
Caproic (normal) X	0.00187	0.2529	0.74
Lauric	0.006	0.1488	0.40
Myristic	0.0713	0.1360	52.40
Arachidic	0.0536	0.0992	54.00
Erucic	0.0468	0.0942	49.70

X Color change with methyl orange gradual, not sharp.

A Test for Castor Oil

It will be seen that there is no appreciable hydrolysis in solutions of sodium salts of the fatty acids, up to, and including lauric acid, but after this there is a complete break, the hydrolysis alkali in the soap of myristic acid and all higher acids amounting to approximately 50% of the total. Rosin acids behave similarly to fatty acids as shown in the table given below.

Briefly the test suggested for distinguishing castor oil from other oils and detecting adulteration is as follows: 5 grams of the oil are saponified by heating with slight excess of aloholic caustic potash solution, under a reflux condenser, phenol-phthalein is added, the liquid exactly neutralized with hydrochloric acid, and the alcohol evaporated off. Water is now added to the residue, and when solution is complete the liquid is cooled, made up to 100 cc., 10 cc. (—0.5 gram of oil) taken, diluted to 250 cc. with boiled distilled water, and titrated with decinormal hydrochloric acid, first in presence of phenol-phthalein and continuing after addition of methyl orange. With castor oil only about 0.5 to 0.8 cc. of decinormal acid was required in the first titration whereas most other fats and oils required 8 or 9 cc. There were, however, a few exceptions, notably butter, coconut, and palm nut fats, the results in these cases being easily explainable when we take into account the presence of the lower fatty acids and know their behavior. Slightly lower results were obtained with sunflower, cotton-seed and linseed oils which are not so easily explainable except on the assumption that they contain some hydroxylated fatty acids.

Since 95% of the acids of castor oil are composed of ricinoleic acid, a hydroxylated acid, it is evident that the salt of this fatty acid does not hydrolyze in solution to any considerable extent, and the difference in behavior between this soap and other soaps must be ascribed to the presence of this particular hydroxylated acid.

The value of the method is shown by the following tests on mixtures of castor oil and arachis oil:

Castor oil Parts	Arachis oil Parts	0.1NHCl required C.c.	Free KHO per 100 of Oil parts	Arachis oil calculated Per cent
100	0	0.5	0.56	...
90	10	1.4	1.57	9.9
80	20	(1) 2.3 (2) 2.3	2.58	19.8
70	30	(1) 3.2 (2) 3.3	3.58	29.7
60	40	4.3	4.81	41.7
50	50	5.0	5.60	49.5
40	60	6.1	6.83	61.5
30	70	6.95	7.78	70.9
20	80	7.8	8.73	80.2
10	90	8.7	9.74	90.1
0	100	9.6	10.75	...

The amount of free alkali found by titration, compared with the total combined alkali, was 3.1% in the case of castor oil and 56.7% in that of the arachis oil. A number of oils and fats have been examined by this method with the following results:

	Free KHO A	Total KHO B	A × 100 B
Castor oil, medicinal.....	0.672	18.48	3.6
	0.784	18.59	4.2
	0.78	17.81	4.6
	0.7	17.92	4.4
	0.7	17.92	4.4
Castor oil, medicinal	0.896	18.70	4.8
	0.898	18.93	4.7
Castor oil, medicinal	0.896	18.70	4.8
	0.896	18.59	4.8
Crude castor oil	0.56	18.14	3.1
(Free fatty acids 1.20%)...	0.56	18.14	3.1
	0.56	....	....
Castor oil, best seconds.....	0.896	18.70	4.8
	0.896	18.81	4.8
Castor oil, Madras.....	1.34	18.25	7.3
	1.45	18.48	7.9
Linseed oil	6.16	20.05	30.8
	6.27	20.05	31.3
Cottonseed oil	8.17	20.16	40.6
	8.29	20.16	41.1
Crude cottonseed oil.....	8.96	20.16	44.4
Dark red	9.18	20.05	45.8
(Free fatty acids 3.1%).....	8.96	....	44.4
End point difficult.....	8.62	....	43.0
Sunflower oil	7.73	19.49	39.1
Naphtha extracted	8.29	19.49	42.5
Alkali refined	....	....	....
Arachis oil	10.64	19.94	53.7
	10.76	19.94	54.0
Olive oil, medicinal.....	9.41	19.82	47.5
	9.52	19.49	48.8
	9.18	....	....
Rape oil (crude).....	8.96	17.58	51.0
	8.62	17.69	48.7
Rape oil (crude Jambo).....	8.85	17.70	50.0
(Free fatty acids 1.04%)....	8.74	17.81	49.0
Rape oil (adulterated).....	4.14	13.21	31.4
	4.03	13.33	30.3
	4.81	13.66	35.2
Pure sesamé oil.....	9.41	19.49	48.3
	9.41	19.49	48.3
Refined sesamé oil.....	9.86	19.49	50.6
	9.86	19.49	49.4
Crude sesamé oil.....	9.41	19.49	48.3
(Free fatty acids 2.9%).....	9.63	19.38	49.8
	9.41	....	48.3
Crude groundnut oil.....	9.52	19.26	49.4
(Free fatty acids 0.99%)....	9.63	....	50.0
	10.08	19.26	52.3
Crude Kapok oil.....	10.2	20.16	50.6
(Free fatty acids 5.2%).....	10.63	20.38	51.6
	10.53	20.61	51.1
	10.64	....	51.6
Palm oil	9.52	19.93	47.7
	9.63	20.16	47.7
	9.08	....	....

	Free KHO A	Total KHO B	A × 100 B
Lard	10.53	20.27	51.9
	10.30	20.38	50.6
Tallow	9.74	19.60	49.7
	9.85	19.82	49.7
Pure Premier Jus.	9.86	20.05	49.2
Tallow oil	9.74	19.71	49.2
(Free fatty acids 0.27%)	8.29	18.41	44.9
Pure British cod oil	8.29	18.59	44.6
Pure filtered	8.62	19.49	44.2
No. 1, whale oil	8.62	19.49	44.2
Pure filtered	8.73	19.49	44.8
Seal oil	8.85	19.38	45.7
Butter fat (1)	8.29	22.96	36.1
	8.85	23.18	38.1
Butter fat (2)	9.30	22.06	42.1
	9.18	22.40	41.0
Butter fat (3)	8.40	22.06	38.1
	8.51	22.06	38.6
Butter fat (4)	9.40	22.18	42.4
	9.18	21.95	41.9
Butter fat (5)	9.52	21.84	43.6
	9.30	22.18	41.9
Butter fat (6)	9.40	21.62	43.5
	9.40	21.73	43.2
Coconut oil	5.15	25.20	20.4
(Commercial)	4.93	25.65	19.2
Cochin coconut oil	3.92	24.98	15.7
(Free fatty acids 1.30%)	4.03	24.86	16.1
French Cochin coconut oil	3.92	24.75	15.8
(Free fatty acids 1.97%)	3.92	24.64	16.0
Ceylon coconut oil	4.59	25.09	18.3
(Free fatty acids 4.57%)	4.37	25.09	17.4
Pure coconut oil	3.81	24.42	15.6
(Free fatty acids 1.00%)	3.70	24.19	15.3
	3.92	....	16.2
Palm nut oil	5.93	25.87	22.9
(Commercial)	5.82	25.65	22.7
Palm nut oil	5.15	23.74	21.7
Alkali refined	....	....	....
(Free fatty acids nil)	....	....	....
Palm nut oil	5.26	24.53	21.5
(Free fatty acids 3.55%)	5.49	24.64	22.3
Palm nut oil	5.60	25.20	22.2
(Free fatty acids 4.93%)	5.49	24.86	22.1
Rosin	9.52	17.81	53.5
	9.52	18.03	52.8

Experiments were conducted with other indicators besides phenol-phthalein and methyl orange; e. g., thymol blue and alizarin red S., which, after allowing for the slight difference in pH for the color change, gave comparable results.

The author is indebted to Mr. John Hanley, F. I. C. and Messrs. Job Ivory & Co., for several of the samples of oils used in these investigations.

Chemical Problems in the Manufacture of Soap

Fundamental Principles of Soap Making Discussed

THE study of soap-making from a chemical point of view presents many interesting problems. Like many other industrial operations, the manufacture of soap has gradually developed from very rude beginnings, by accumulated experience, to become a highly specialized branch of chemical manufacture.

It was well known that when an oil or fat is mixed with a solution of caustic alkali—soda or potash—and allowed to stand for some little time, at a moderate temperature, a solid product, or soap, is formed. This, in short, was the "cold process" of producing soap, but in this operation any excess of alkali, also any impurities contained in the alkali and the fat, as well as the glycerine, remained with the soap, which was, therefore, a variable and far from pure product. It was also known that saponification could be accelerated by heating the oil and alkali solution and bringing them into intimate contact by agitation, and when it was discovered that the soap could be "salted out" or separated by addition of common salt, the removal of impurities, together with the glycerine, become possible, and better qualities of soaps could be produced.

These operations of the soap maker, although simple in appearance, depend for their success upon an intimate knowledge of the raw materials and the careful adjustment of the working conditions, since without such knowledge and attention to details it would be a matter of luck if a satisfactory finished product resulted, and the regular production of a standard quality of material would be altogether out of the question.

Composition of Oils and Fats

The composition of fats and oils was established on a scientific basis by the researches of Chevreul, described so clearly and accurately in his remarkable work, "Recherches Chimiques sur les Corps Gras d'Originie Animale," 1823. He showed that when a fat or oil is heated with an alkali two very definite products are always formed, viz., a soap and glycerine (the latter previously discovered by Scheele, in 1779, by heating lard with lead oxide and water in the preparation of lead plaster). He proved that fats are combinations of fatty acids with glycerine—or, rather, with its radicle glyceryl—and that on heating with alkali the latter combines with the fatty acids to form salts, or soaps, while thus replacing the glycerine which appears in the free state.

Chevreul was able to identify several fatty acids in various oils and fats; he separated and analyzed these acids and examined many of their compounds, and he also demonstrated that natural oils and fats are composed of mixtures of the simple glycerides, olein, stearin, etc., in various proportions; the nature and relative quantities of these governing the properties of the product.

On separating the two constituents—fatty acids and glycerine—and weighing them, it was found that their combined weight was greater than that of the original fat taken, which is explained by the fact that the elements of water are taken up during the reaction, and he concluded that saponifiable fats have a composition exactly similar to that of the compound ethers, the alcohol in this case being glycerine, which is capable of uniting with fatty acids with the elimination of water and the formation of the esters (triglycerides or fats).

The conclusions arrived at by this great investigator 100 years ago have been amply confirmed by numerous later workers, who have also added considerably to our knowledge. Berthelot extended the work, and showed that fats can be produced artificially by the direct union of fatty acids and glycerine under heat and pressure with elimination of one, two, or three molecules of water and the formation of what we now know as mono-, di-, and tri-glycerides, respectively.

Although it is possible to prepare these three kinds of glycerides artificially, the glycerides found in natural fats are, probably without exception, entirely those known as tri-glycerides, i.e., triolein, tripalmitin, tristearin, etc., or mixed triglycerides, such as oleo-dilaurin.

From the different characters of the various oils and fats it is evident that they do not all possess the same composition; in fact, there are no two alike, and no natural fat or oil consists of only one glyceride; usually four or five glycerides are present; sometimes even more. Perhaps the nearest approach to a pure glyceride is castor oil, which contains nearly 95 per cent. of triricinolin. Recent investigations have however, shown that the glycerides are compound, i.e., two or three fatty acids may be in union with the same molecule of glycerine, forming what are known as mixed glycerides, and quite a number of these compounds have been isolated and examined. The possible number of these molecular compounds is infinite, and their influence upon the properties of oils and fats has only recently been realized.

The crude oils and fats are never neutral, a proportion of free acids being always present. The amount of free fatty acids depends upon the nature of the original raw material, and also upon the method of extracting the oil. Animal fats and oil usually contain little free acids, but vegetable oils often contain a very large proportion; thus, the free fatty acids in tallow rarely exceed 5 per cent., whereas olive oil sometimes contains 25 per cent. and palm oil may contain as much as 80 per cent.

Fat-Splitting Enzymes

If the original raw material is damp or mouldy then the oils extracted therefrom contain a high percentage of fatty acids, the latter being liberated by the enzymic action of moulds and bacteria, the glycerine liberated at the same time being to a large extent destroyed, with consequent low yield of that commodity in the glycerine extraction house. Several seeds contain also active principles known as "lipolytic" or fat-splitting enzymes, which are liberated when the seeds are crushed, and these, assisted by moisture, warmth, and a slight acidity, are capable of converting neutral fats into fatty acids and glycerine completely, and in very short

time. Castorseed is a notable example of a seed containing a lipolytic enzyme or "lipase," the application of this seed in a crushed state for fat-splitting being the basis of several patents by Connstein, Nicloux, and others. Palm fruit pulp and olive pulp also contain fat-splitting enzymes, which accounts to some extent for the high percentages of fatty acids in the oils from these sources, but these oils are to a large extent prepared from more or less decomposed material, in which moulds and bacteria also generate enzymes of a similar character.

Unsaponifiable Matter

The unsaponifiable matter in fats and oils, consisting, for the most part, of cholesterol or phytesterol and their esters, plays no definite part in soap-making, since the amount present is so small, in most cases, rarely amounting to more than one per cent.; but certain oils contain high proportions of unsaponifiable matter. For instance, sperm oil contains about 45 per cent. of alcohols, which appears as unsaponifiable matter, and certain shark liver oils have been found to contain unsaponifiable hydrocarbons in such quantities as to render them entirely unsuitable for soap-making.

It is a well-known fact that in the manufacture of soap by the boiling process strong solutions of caustic soda are unsuitable in the first stages, since the oil and alkali do not mix; that is, the interfacial tension between the two is so high that there is practically no interpenetration, any action which takes place being possible only at the surfaces in contact or by violent agitation; the strong soda solution, behaving in a similar manner to common salt in salting out any soap which may be formed, also interferes with the saponification. It is, therefore, necessary to employ weak solutions of alkali at first through which the oil can be disseminated in a finely divided condition in the form of an emulsion. In emulsions the oil globules are reduced to extremely minute dimensions, and thus they expose an enormous surface to the hydrolytic action of the alkali. At the same time, the soap formed lowers the interfacial tension between the oil and the water, so that the oil globules are still further comminuted and saponification proceeds more rapidly, the soap, in this case, remaining in solution, the alkali is free to exert the maximum action consistent with the conditions.

In this direction, the presence of free fatty acids in the oil is a distinct advantage, since the combination of these with the alkali is almost immediate, and no external energy is required to bring it about; the saponification of the oil is thus accelerated by their presence.

Rate of Saponification

The rate at which saponification proceeds will vary with the nature of the oil or fat used, the temperature, and the strength of the solution of alkali. All fats and oils are not equal in the ease with which they can be saponified. Coconut oil is very quickly saponified, and so is castor oil, but olive oil not so readily and tallow still less easily; but by blending oils which are readily saponified with those that are not, the saponification of the mixtures is rendered easier.

The question has arisen as to whether certain glycerides in oils are saponified before others, or whether the oil is saponified as a

whole, and also whether the saponification, once started, proceeds direct to the formation of soap or in three stages, with the formation of diglyceride, monoglyceride, and then soap. These problems will have to be dealt with later.

There is every reason to believe that some glycerides are more readily saponified than others; the glycerides of the lower fatty acids, for instance butyric, caprylic, etc., up to myristic acid, appear to be more readily saponified than the glycerides of the higher acids, e.g., palmitic, stearic, etc., but they are so combined with one another in the natural fats that the individual glycerides are not selectively hydrolyzed, the fats being apparently saponified as a whole.

In the manufacture of commercial soaps it is usual to blend two or more oils together to obtain particular results.

Tallow or stearin forms an extremely hard soap, which does not readily dissolve in water, forms a stiff paste with water, and does not readily lather, although the lather, once formed, is a lasting one. On the other hand oils usually yield softer soaps which are more easily soluble, form thin solutions, and readily lather, although the lather is not very permanent. By blending tallow with oils a fairly hard soap is obtained, which dissolves moderately easily in water, forms a thicker solution, which lathers easily, and the lather is more permanent.

Palm oil forms a soap which is intermediate in its properties between that of tallow and the oils, and forms a useful third material for blending the others together.

It may be noted that oils, such as olive oil, containing fatty acids belonging almost entirely to the oleic series form very good hard soaps, whereas those containing a high percentage of the more unsaturated acids—linolic, etc.—i.e., cottonseed oil, linseed oil, fish oils, etc., form pasty soaps, and are unsuitable to be used alone, except for soft-soap making. Castor oil forms a hard soap, but the solutions are very thin, it gives very little lather, has very little detergent properties, and does not hydrolyze like other soaps when dissolved in water.

The Quantity of Alkali

It has been stated that saponification is never complete unless the alkali is in excess, but this is not correct; complete saponification can be brought about with the theoretical quantity of alkali, provided the right conditions are ensured. Excess of alkali is more likely to retard saponification than is a deficiency. In cold saponification, in which a strong solution of alkali is stirred into the warm oil and the mass kept at a temperature of 70° to 80° F., 95 to 99 per cent. of the oil will be saponified in 48 hours, and if the temperature of the oil be raised to about 160° F. and the solution of alkali is well stirred in, the saponification is so rapid that the whole mass will set to a stiff translucent jelly in about half an hour.

Salting Out

After the saponification is almost complete, we have in the boiling process a mixture consisting of a solution of soap and glycerine, some free alkali, and some unsaponified fat. Since soaps are hydrolyzed by water, there will be free alkali and an acid soap, or perhaps free fatty acids. On addition of common salt the soap becomes much more viscous, and at a certain concentration of salt two layers are formed, a concentrated solution of

soap on the surface containing the unsaponified fat and a salt solution below containing the glycerine. There may be a third intermediate layer, containing a proportion of all the constituents. If the salt is added in sufficient quantity soap curds will be formed. This action takes place not only with strong solutions of soap, but with comparatively weak ones also. Caustic soda has the same effect as common salt, and is even more effective; it also forms curds. Carbonate of soda and sulphate of soda also will salt out soap, but they are not so effective, and they behave in a somewhat different manner, forming two liquid layers, the upper soap layer setting to a gelatinous mass on cooling. Sulphate of soda is the least effective.

Effect of Salt

The effect of the salt is, however, not merely that of separating the soap in the form of a paste or as curds. There are in the kettle, besides neutral soaps, the products of dissociation—free alkali and an acid soap or free acids—which are caused to recombine by the addition of the salt; that this is the case may be demonstrated by estimation of the free alkali before and after such addition.

The writer has been able to touch upon only a few points in the present article. As time goes on, other problems will be dealt with, and, no doubt, there will be opportunities for treating upon these subjects in a fuller manner. *Oil and Color Trades Journal.*

The Detection of Extracted Olive Oils

Organic Silver Salts Exhibit Marked Reactivity to Sulphur

By M. F. LAURO

THE various tests for extracted oils are based on reactions with traces of the solvents left in the oils after extraction. On Olive Oil Foots or Industrial Olive Oil, extracted from olive pulp by means of carbon bisulphide, sufficient solvent is always present to be easily ascertainable, by almost any of the methods used for the detection of sulphur or sulphur compounds. When, however, such oils are bleached, refined and blended with pressed oils, the percentage of solvent present is considerably reduced, generally to a point where it becomes quite a problem for the chemist to determine its presence.

Metallic Silver Method

The so-called "Coin" test is the one most often used in the United States for the detection of carbon bisulphide in olive oil. The suspected oil is heated, with a bright new dime or piece of silver foil suspended half-way in the oil, in a test-tube immersed in an outer oil bath to prevent scorching. The heat is gradually carried up to 210°C. taking from fifteen to twenty minutes, then held at that temperature for another fifteen minutes. The silver is examined to see if blackened. A black stain on the silver indicates the presence of sulphur or its compounds. At times the metal is stained a coppery hue, rather than black, in

which case the oil may be further heated to 240°C. and the color noted. Some olive oils may discolor silver but unless the tarnish is black on the coin, even though slight in extent, the test is not considered conclusive.

Acetic Anhydride Reaction

In Italy, a test frequently used is that of Pachini or Bracci, where a few cc. of oil are warmed with acetic anhydride, filtered through a paper wet with the anhydride, and a drop or two of concentrated sulphuric acid allowed to come in contact with the filtrate. A rose-red coloration changing to violet, or to green when diluted with water, indicates an extracted oil.

Organic Silver Method

In place of metallic silver, which is not sensitive enough and which requires a temperature near the scorching point of olive oil, the writer employs an organic salt of silver, such as the oleate, salicylate or preferably benzoate, in finely powdered form.

The test is conducted as follows: About five cc of the oil to be examined is heated in a test tube to 150°C., and a pinch of silver benzoate is dropped in from a spatula and shaken into the oil. If any sulphur-bearing solvent be present, the oil immediately begins to darken and will discolor proportionately to the percentage of sulphur present. If a mere trace is present the benzoate may be added to the oil in the beginning and then heated, due care being taken to avoid scorching of the oil.

This test has been found sensitive to 0.2% of olive oil foots in a pressed oil, as determined by comparing the color formed with a blank of the original oil without addition of "foots," but heated with benzoate, in the same manner. Neither the "coin" nor "Pachini" test reveals the presence of this amount.

Physical Changes

Concurrently, the writer makes use of the color change and the odor of olive oil on heating as confirmatory tests. A good edible oil will have a characteristic odor, somewhat like garlic, and in most cases the color changes on heating from golden yellow to a distinct light green. In heating extracted oils, not only does the color darken with silver benzoate to reddish black and black, so that it is opaque, but the odor is decidedly unpleasant and "choky." With commercial pressed oils, the color may darken slightly, which sometimes happens, but the oil is not opaque.

Oil extracted with ethylene trichloride now appears often on the market. The Beilstein copper-wire test easily shows the presence of this halogen by the green coloration imparted to a flame when an ignited copper wire wet with the oil is thrust therein. Such oils lighten, rather than darken in the benzoate test.

Since many compounds that affect silver salts, such as aldehydes, are present in inferior, rancid and high acid olive oils, there is at times a slight reduction of silver with such oils, but the color change is slight under the conditions of the test and easily differentiated from that with oils containing carbon bisulphide.

If 0.02 gram of silver benzoate and 5 cc. of oil are used, the test may be made quantitative by comparing the color produced against that shown by standards containing known amounts of olive oil foots or of carbon bisulphide made up and run at the same time.

The Behavior of Fish Oils With Uranium Nitrate and Pyrogallic Acid

Developing a Possible Method for Detection of Adulteration in Cod Liver Oil.

By WALLACE H. DICKHART

THERE has been considerable desire on the part of importers and dealers in Cod Liver Oil for the development of a means of determining the purity of that commodity which would be, (while reasonably dependable), more rapid than the present method prescribed by the U. S. Pharmacopoeia, which requires the determination of unsaponifiable matter, saponification and iodine numbers.

The following data, which was assembled while the writer was conducting experiments with a view to obtaining a chemical test for the identification of vitamins, seems to offer an avenue for research, which may, upon further investigation, develop a rapid means of confirming the purity of Cod Liver Oil, within reasonable limits.

Reagents Selected

Uranium Nitrate and Pyrogallic Acid were used for the following experiments, because, first of all, uranium nitrate being a radio-active chemical, was considered a likely substance for the development of a chromogenetic reaction. According to the U. S. Dispensatory, it is incompatible with the alkaloids, chloral hydrate, salts of arsenous and phosphoric acids; and changes the color of cochineal and its derivatives. The oxide is frequently used in photography. Pyrogallic acid was chosen because of its reaction with trichloracetic acid and kerosene in the detection of vitamins and also because of its use in photography as a developer.

Variations of Vitamin Tests

Knowing that pyrogallic acid when used with trichloracetic acid, kerosene and hydrogen peroxide, will develop a pink coloration with the vitamins of Cod Liver Oil,[1] I substituted powdered uranium nitrate for the trichloracetic acid, but the results were negative. When, after experimenting for some time, a mixture of equal parts of the uranium nitrate and pyrogallic acid were powdered together in a mortar, one-tenth of a gram of this mixture placed in 5 cc of oil in a test tube and shaken without heat, no reaction resulted; but on heating the test tubes over a Bunsen flame a color developed much more quickly with the other oils than with Cod Liver Oil. This appeared to be a good lead for a cod liver oil purity test and each chemical used was tested separately for its solubility in the various fish and marine animal oils worked with. Both the uranium nitrate and pyrogallic acid were soluble in all the oils tested, which included cod liver oil, menhaden oil, pilchard oil, whale oil, sperm oil, sardine oil and herring oil.

Experiments on Characteristic Reactions

It was this stage of the experiment that led to more encouraging

[1] Am. Jour. of Phar., July, 1926, p. 422.

results. In the first series of tests 0.1 gr. of pyrogallic acid was added to 5 cc of each of the oils, followed by a trace of powdered uranium nitrate. This mixture was placed in the steam bath for a few minutes and occasionally shaken. With all the oils, other than cod liver oil, a precipitate was formed and the color of the mixtures became a very dark red. With the cod liver oil, the mixture assumed a medium red color but no precipitate appeared. In the next experiment 2 grs. of pyrogallic acid were dissolved in 100 cc of ether; 5 cc of this solution were added to 5 cc of each oil in a test tube and the tubes placed in a water bath at 45°C. to evaporate the ether. After the ether was evaporated, 10 milligrams of powdered uranium nitrate were added to each oil, and the mixtures placed in the steam bath for thirty minutes, after which they were removed and the colors compared before an electric lamp. All the oils had turned a dark reddish or a brown color and were opaque, except cod liver oil, which although of a light red color was translucent. The ether solution of the pyrogallic acid was found to be an undesirable reagent, as it turned red on standing.

Another experiment was conducted by making a 1 percent alcoholic pyrogallic acid solution, using 95% alcohol. It was necessary to use 95% alcohol, as pyrogallic acid in weaker alcoholic solution turns yellow on standing. In this experiment a reversal of the procedure in the experiment above was adopted.

Ten (10) milligrams of the powdered uranium nitrate were placed in a test tube with 5 cc of the oil. The tube was placed in the steam bath for about two minutes to dissolve the uranium salt. Then 5 cc of the alcoholic pyrogallic acid solution was added and the mixture heated until most of the alcohol had evaporated. The tube was then removed from the bath to cool. The following colorations in the tubes resulted:

Oil	*Primary coloration*	*Treated with alcohol and sulphuric acid*
Cod Liver Oil (U.S.P.)	Orange Red	Decolorized
Harden Fish Oil	Orange Red	Decolorized
Sperm Oil	Orange Red	Decolorized
Sardine Oil	Yellowish Brown	Yellow
Whale Oil	Yellowish Brown	Yellow
Menhaden Oil	Yellow	Canary Yellow
Pilchard Oil	Yellow	Yellow
Newfoundland Cod Oil	Orange Red	Bright Orange
Herring Oil	Light Orange Red	Yellow
Blank Determination	Red	Decolorized

Cod Liver Oil (U.S.P.) orange red
Hardened Fish Oil orange red
Sperm Oil orange red
Sardine Oil yellowish brown
Whale Oil yellowish brown
Menhaden Oil yellow
Pilchard Oil yellow
Newfoundland Cod Oil orange red
Herring Oil light orange red
Blank Determination red

Next 10 drops of a 1-4 mixture of alcohol and sulphuric acid were

added to each tube, and the contents well shaken, with the results in table on page 327.

A further variation of the experiment was conducted as follows: Ten milligrams of the powdered uranium nitrate were placed in a test tube with 5 cc of the oil. The tube was placed in the steam bath to dissolve the uranium nitrate. One-tenth of a gram of pyrogallic acid was added and the mixture again heated in the steam bath for two minutes. All the oils except cod liver oil gave a precipitate and a color. They were allowed to stand overnight and in the morning examined to observe the appearance of the precipitate under the microscope. By this time the cod liver oil had precipitated white crystals which had the appearance of branches of trees each with a number of small twigs extending from the branch. Whale, menhaden and sardine oils gave a different form of crystal. In these three oils the crystals were more of a disc-mould or jelly-fish shape with many fine hairy legs and at the end of each leg a small ball. These objects were different in appearance from the fatty acid crystals which could be seen throughout the slides. Herring Oil gave a needle-shaped crystal, while Pilchard Oil gave crystals which looked more like staphylococcus bacilli. The original oils were tested to see if moulds were present but none was found.

Proposed Test for Cod Liver Oil

The following simple experiment can, I believe, be used to differentiate between cod liver oil (U.S.P.) and contaminated cod liver oil. Directions: Place in a test tube 10 milligrams of powdered uranium nitrate and 3 cc of the fish oil to be tested, then place the tube in the steam bath for 20 minutes, shaking occasionally, remove and observe the color, which should be as follows:

Cod Liver Oil (U.S.P.)—Amber color, showing a greenish cast with transmitted light
Norwegian Sperm Oil—Light amber color, no difference with transmitted light
Menhaden Oil—Crimson red
Pilchard Oil—Light red
Whale Oil—Light brownish red
Herring Oil—Blood red
Sardine Oil—Blood red
Newfoundland Cod Oil—Blood red

Samples of cod liver oil that were thought to be pure, answering all the U. S. P. requirements, except that they were high in unsaponifiable matter, gave a red color within six minutes. These samples were later declared contaminated.

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Tangible Profits Accrue to Millers and Refiners Through Improvement of Quality

By JOHN P. HARRIS

IN a previous issue of OIL & FAT INDUSTRIES, the writer has outlined the advantages to the edible oil refiner of using a purely absorptive activated carbon for bleaching purposes, either alone or in combination with fullers' earth or other bleaching agents. In that article the economies of bleaching refined oil with such carbon were stressed and inasmuch as this carbon acts particularly well in the removal of all impurities from refined oils, even incuding traces of fatty acids, it naturally occurred to us to try it out upon the crude oil itself, particularly since it works as well cold as it does hot, as it is surely logical to attack the removal of color, odor, flavor and all of the emulsificants present in crude oil, right at the start of processing, whereby their presence will not affect the subsequent stages of refining.

For example, in a prime crude cottonseed oil, possessing under 1% free fatty acids, we invariably have a neutralizing loss of considerably more than five times the free fatty acids. If we were able to free the crude oil of *all* of the emulsifying agents, pigmentation, etc., by completely cleansing the crude oil, we could neutralize that oil with a loss very close to the percentage of free fatty acids.

It Produces Almost Theoretical Losses Upon Coconut Oil

This we have already accomplished in the case of coconut oil, where, for example, by treating crude coconut oil running 5% free fatty acid (oleic), in the cold with a purely absorptive carbon, we have been able to reduce the neutralizing loss to 5.5%, and we can accomplish such results consistently, and with varying percentages of free fatty acids in the crude oil.

A Great Benefit in Treating Crude Linseed Oil

Experiments were started some years ago, covering the purification of crude linseed oil with this purely absorptive carbon. The results showed that a better varnish oil could be obtained by first treating the crude oil with carbon prior to neutralizing, than by neutralizing first and using a bleaching agent thereafter.

Excellent results have also been obtained in the pretreatment of crude peanut oil with this purely absorptive carbon.

But only recently have we had the opportunity of trying this out upon the economically most important vegetable oil—American Cottonseed Oil.

Advantages of Pre-treating Crude Cotton Oil

We shall cite here two, from a series of large scale tests, made in conjunction with an American crusher and refiner. In these tests, the oil was followed through

from the crude mill, to the deodorizer and finishing press, and the crude oil was first filtered as it came from the presses, so that the advantages of straight filtration should be eliminated as far as possible, in order that the results might represent exclusively the effect of the purely absorptive carbon only, upon the crude oil.

This carbon treatment consisted of adding to the crude oil, ¼% of this purely absorptive carbon, together with ¼% of diatomaceous earth, as a filter aid, the latter being used to insure satisfactory, quick and easy filtration at room temperatures. This mixture was agitated at 28°C. for thirty minutes and was then pumped through a recessed plate filter press. The cake was blown and and steamed, and the residual oil determined in the laboratory. Results were as follows:

Test I. *Untreated Crude*
10% of 15° Be. Caustic Soda used in refining.
Refining loss 8.1%
Free fatty acid 2.9%
Flavor and odor Sour
Grade off.

Carbon Treated Crude
Same amount of Caustic Soda used in refining.
Refining loss 5.95%
62% of oil in press cake, therefore loss of oil in press cake equal to ½% x 0.62 or 0.31%. Net Reduction of Refining loss 1.84%.

Test II. *Untreated Crude*
Free Fatty Acid 3.54%
Flavor and odor Sour
Grade Off
Refining loss 10.60%

Carbon Treated Crude
Same amount of Caustic Soda used in refining.
Refining loss 7.10%
Percentage of oil in the press cake was the same as in Test I.
Net Reduction of Refining Loss 3.19%

The average net reduction of refining loss in the two instances quoted was 2.51%. Figuring on the present market value of oil and allowing for soap stock, this would mean a saving of about $90.00 on every 160 barrel tank car treated.

Color of Oil

The results obtained in the above tests are sufficiently striking to merit particular discussion. The refined oils obtained without treatment of the crudes other than straight filtration when hot, were further treated with a total of 0.9% of the bleaching agents commonly employed by this refiner, and the color was satisfactory.

The refined oil obtained from the carbon-treated crude, however, was lighter and brighter *without any further treatment*, than the refined and bleached oil from the untreated crude.

This represented a further saving, for the costs incidental to bleaching would amount to about ½% of the value of the oil, or about $24.00 based on a tank car.

Deodorization of Oil

The bleached refined oil obtained from the untreated crude, and the refined oil from the treated crude were then sent on to the deodorizer and the difference in duration of time necessary to deodorize was noted. The oil from the carbon treated crude enabled this refiner to cut the deodorizing period by 45 minutes per batch, lessening his steam consumption by 15%, and increasing his daily output capacity proportionately.

The actual steam saving would come to about $2.00 per tank car treated, in their case.

Status of Meal and Cake Grading

A. S. RICHARDSON, Chairman of the Meal Grading Committee writes as follows:

"Although we are making progress in the study of meal grading, a large proportion if not all of the new season's meal will be graded before any official action can be taken by all the committees concerned with the development and adoption of the method.

"A search through the various journals suggests that just enough has been published on this subject to be confusing and this idea is further confirmed by several letters which I have received. Therefore we believe that a statement should be published in OIL & FAT INDUSTRIES, explaining the present status of meal and cake grading.

"I have taken the matter up with the other members of the committee on method of color comparison and we have prepared a statement which we enclose herewith in duplicate. Although it is merely a statement of the present situation and not a new committee report, we suggest that you pass it along for publication as a news item."

Interstate Rule 102 provides that prime cottonseed cake and meal of 41 per cent or higher protein content shall not be darker in color than Munsell Color Standard "2 yellow 6/5" and that 36 per cent protein cake and meal shall not be darker than Munsell Color Standard "10 yellow 5/5."

The Interstate rules do not yet provide a specific procedure for making the comparison between color standard and sample of meal or cake. In November, 1926, a special A.O.C. S. committee proposed two such procedures in a report which was subsequently published in the May 1927 issue of OIL & FAT INDUSTRIES (pages 187-8). Neither of these methods was definitely adopted, although it was the consensus of opinion of the Chemists' Committee of the Interstate that the simpler method (reproduced below) was preferable as a tentative method for the remainder of the 1926-7 season.

The special committee for study of methods of color comparison has been continued for another year and the adoption of an official procedure is awaiting the further study of that committee. In the absence of an official method, the most satisfactory procedure for general use will be method II of the report in the May issue of OIL & FAT INDUSTRIES. Method I of the same report, based upon the use of a rotating cup, has been adopted by the Texas Cotton Seed Crusher's Association in Rule 271, Supplement No. 1, 1927-8. The rotating cup method is believed to give more satisfactory results in doubtful cases but is less simple of manipulation. Method II, which was tentatively favored by the Chemists' Committee of the Interstate last season, is as follows:

"a. Meal. The meal to be graded should be placed in the center of a gray sheet or board at least eight inches square; it should be flattened out to make a level circle about three or four inches across, and a clean, one-inch square of the color standard laid on the center of

the meal. The meal and standard, lying in a horizontal plane, should then be observed, in good daylight, from a position directly above them and at least 36 inches distant. For making close decisions, it is best to lay the board on the floor and observe it from a standing position directly above. To be graded "prime," the meal must be as light or a lighter shade than the standard. If darker, it must be graded "off" in color.

"b. Cake. A representative portion of the cake to be graded should be ground so that 85 per cent will pass a 20-mesh screen.* Portions of sample used for screen test should not be used for color comparison. The ground sample should be graded as for meal.

"Note: Any samples of meal containing coarse particles should be ground to the standard for cake and this fact should be stated in the report."

* The committee report as published adds the words: "and 75% through 30 mesh." The committee has since concluded that the double mesh specification is objectionable.

Grading Standard Glasses

To All Members:

The Governing Board has employed Miss Geraldine K. Walker, formerly of the Munsell Color Co., as "Research Associate" at the Bureau of Standards to conduct "An Investigation into the Uniformity of the Lovibond Glasses at present in use in the Vegetable Oil Industry."

As a part of that work it will be necessary to grade all glasses submitted, and in due time Mr. A. W. Putland, who has consented to handle the matter will invite the members to send him any glass which they wish graded at $1.50 a glass. He will issue A. O. C. S. certificates of the value of each glass submitted.

We feel that this is a step in the right direction and that the results of this work will be of great benefit to the industry as a whole.

H. P. TREVITHICK,
President.

F. F. A. Co-operative Tests

This co-operative work has been conducted during the past season by the Refining Test Committee. The main object was to stimulate interest in getting correct F.F.A. tests because of their importance in refining work. The F.F.A. test is the first step in refining a crude oil, and if incorrectly made it will result in selecting an improper lye for refining, and this in turn will lead to improper refining results. Ten samples were sent out. The first went to nineteen laboratories, number two to twenty-nine, and the balance to thirty-two laboratories. Interest, however, fell off to such an extent that while all nineteen laboratories reported on the first sample, not more than twenty out of thirty-two reported on any of the last three samples.

The accompanying table shows the total "points off" from the accepted average for each laboratory. A tolerance of two hundredths per cent from the accepted average is allowed in all cases, the same as in the Smalley Foundation work. This means that any test within .02% of the accepted average is considered perfect.

The names of the five laboratories making the best showing together with the average "points off" are also shown in a separate table. Laboratory No. 10 at the head of the list reported on eight

samples, and on three of these obtained perfect results by both brine and alcohol, and was also perfect on brine in a fourth sample, and perfect on alcohol on a fifth sample, thus scoring eight perfect results out of a possible sixteen. The other four laboratories on this list each scored from four to nine perfect results. All five of these laboratories averaged less than .05% off from the accepted average.

Our conclusions from this season's work are as follows:

(1) There is altogether too much variation among the different laboratories for such a simple test. This indicates a lack of care. There is no reason why all laboratories cannot average less than ten points off by using proper care.

(2) Alcohol gives slightly higher results on every sample in hands of every operator. The average of all ten samples shows .12% higher.

(3) Alcohol shows more concordant results among the various laboratories than brine on every sample without exception.

C. B. CLUFF.

Total "Points Off" from "Accepted Average"

A "tolerance" of ± .02% from "accepted average" allowed in all cases

	Brine		Alcohol		Combined		
Lab.	Samples Reported	Points Off	Samples Reported	Points Off	Sample Reported	Points Off	Av. Pts. Off
1	4	20	8	61	12	81	6.8
2	9	173	9	161	18	334	18.5
3	9	228	9	230	18	458	26.1
4	5	114	4	55	9	169	18.8
5	9	50	9	22	18	72	4.0
6	7	68	7	57	14	125	8.9
7	10	94	10	69	20	163	8.2
8	3	33	3	33	6	66	11.0
9	9	28	9	39	18	67	3.7
10	8	15	8	38	16	53	3.3
11	10	122	10	61	20	183	9.2
12	10	112	10	155	20	267	13.4
13	8	194	8	69	16	263	16.4
14	9	96	9	69	18	165	9.2
15	7	112	8	26	15	138	9.2
16	4	80	4	43	8	123	15.4
17	9	67	9	63	18	130	7.2
18	9	67	9	45	18	112	6.2
19	7	160	7	52	14	212	15.1
20	9	192	9	100	18	292	16.2
21	2	23	2	4	4	27	6.8
22	3	24	3	9	6	33	5.5
23	3	52	3	48	6	100	16.7
24	5	122	3	87	8	209	26.1
25	9	517	9	526	18	1,043	57.9
26	8	90	8	80	16	170	10.6
27	1	24	1	9	2	33	16.5
28	7	159	7	89	14	248	17.7
29	8	44	9	34	17	78	4.6
30	1	32	1	25	2	57	28.5
31	7	21	7	26	14	47	3.4
32	7	201	7	159	14	360	25.7

Best Laboratory Work

Consideration given only to laboratories reporting on at least 70% of the samples sent out

Order	Lab. No.	Name	Samples	Av. Pts. Off
1	10	P. & G. Co., Staten Island, N. Y.....	16	3.3
2	31	P. W. Tompkins, S. F., Col.....	14	3.4
3	9	P. & G. Co., Ivorydale, Ohio.....	18	3.7
4	5	H. P. Trevithick, New York.....	18	4.0
5	29	Lever Bros. Co., Cambridge, Mass....	17	4.6

The Editor,
OIL & FAT INDUSTRIES.
Sir:

Mr. Chapman's invitation for correspondence on his article in the May issue of OIL & FAT INDUSTRIES brings this from me, since accuracy of results cannot be overestimated.

In Mr. Chapman's directions under the heading of "Determination" the addition of the small piece of paraffine before digestion, that he calls for, is entirely useless, if not detrimental. Its effect is to retard the oxidation of the sample and to deposit carbon in the neck of the digestion flask.

The directions to digest only 45 minutes leads to low results as all the nitrogen of the protein has not by this time, been converted into ammonia. If the digestion is stopped at the end of 45 minutes, only 94% to 97% of the total protein nitrogen has been converted into ammonia, and the report of the protein content is correspondingly low. Where 50 to 80 samples were run per day, the time of digestion being 1 hour and 10 minutes, fully one-third of the samples failed to check within 0.3% protein.

It takes a digestion of an hour and a quarter under the best regulated conditions to effect the change of all the protein nitrogen into ammonia, and where some burners do not give as hot a flame as others in the bank, the digestion should be carried on for an hour and a half. After the boiling acid begins to clear, the heat should be increased until the condensing ring is about an inch up in the neck of the flask.

Guard against the use of too much potassium (or sodium) sulfate. Five grams of potassium sulfate will give the same results as ten grams, but if more than ten grams are used with twenty-five cc of sulfuric acid, a slightly lower percent of nitrogen will be found.

Another caution, do not use such accelerators such as permanganates or perchlorates, as is sometimes recommended. These will oxidize some of the ammonia and lead to low results. Very truly yours,

PAUL L. MENAUL.

The Editor,
OIL & FAT INDUSTRIES.
Sir:

I have received Mr. Menaul's letter regarding the publication of my method for protein in your May issue. I have checked this method many times but in order to give you some late comparative figures I had our Mr. John T. Scott run check meal sample No. 13 four different ways. Probably the sample had dried out slightly as the ammonia was slightly higher than accepted average for this sample.

In making the following determinations the weight taken was 2.5g. The weight of sodium sulphate used 7.0g. Other variables are shown in the table which follows:

Time of Dig.	Mercuric Oxide used grams	Titr. cc		Protein %	Ave. %	Ammonia %
30 Minutes	1.00	5.4	50 — 5.4 =	44.60		
30 Minutes	1.00	5.4	50 — 5.4 =	44.60	44.67	8.69
30 Minutes	1.00	5.2	50 — 5.2 =	44.80		
45 Minutes	.70	5.4	50 — 5.4 =	44.60		
45 Minutes	.70	5.4	50 — 5.4 =	44.60	44.67	8.69
45 Minutes	.70	5.2	50 — 5.2 =	44.80		
75 Minutes	.70	5.4	50 — 5.4 =	44.60		
75 Minutes	.70	5.1	50 — 5.1 =	44.90	44.73	8.71
75 Minutes	.70	5.3	50 — 5.3 =	44.70		
3 Hours	.70	5.2	50 — 5.2 =	44.80		
3 Hours	.70	5.2	50 — 5.2 =	44.80	44.80	8.72
3 Hours	.70	5.2	50 — 5.2 =	44.80		

From the results obtained it will be seen that the difference between 45 minutes digestion and one hour and fifteen minutes digestion is very slight. Using a larger amount of mercuric oxide also decreases the time needed for digestion without any resultant loss of accuracy. Three hours' digestion does however, seem to give better checks but for most purposes the shorter time should suffice. It was not the intention to intimate that all samples should be digested for 45 minutes only. As Mr. Menaul has indicated, flames and other conditions vary and the time of digestion should always be left to the judgment of the operator. We mentioned 45 minutes because we had found that this was about the average for our own work.

We have never found that the use of paraffine had anything to do with the time or completeness of oxidation. With certain classes of material it helps to prevent foaming in the digestion and it a'ways prevents foaming in the distillation, especially if there has been added an excess of caustic soda so'ution. It is merely used as a precautionary measure and if proper quantities of reagents are used, the paraffine may be dispensed with.

Since the chief use of the sulphates of Sodium or Potassium is to raise the boiling point of the digestion there is no object in using an excess. In order to get the greatest efficiency the proper amount must be used.

I agree that the use of accelerators such as mentioned by Mr. Menaul is to be condemned.

I should like to hear from anyone who has suggestions or criticisms to make on this article or on the method.

Very truly yours,
R. M. CHAPMAN.

Referee Applicants

Applicants for Referee Chemist Certificates are Herman A. Nester, San Antonio, Texas, (second publication), and (first publication), A. W. Horrell, Jackson, Miss.

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Second Quarter Census Report

THE Department of Commerce announces that the factory production of fats and oils (exclusive of refined oil and derivatives) during the three-month period ended June 30, 1927, was as follows: Vegetable oils, 487,200,695 pounds; fish oils, 7,008,433 pounds; animal fats, 583,333,175 pounds; and grease, 102,615,614 pounds; a total of 1,180,157,917 pounds. Of the several kinds of fats and oils covered by this inquiry, the greatest production, 458,802,329 pounds appears for lard. Next in order is cottonseed oil with 204,902,831 pounds; linseed oil with 162,232,121 pounds; tallow with 122,178,405 pounds; coconut oil with 71,031,662 pounds; and corn oil with 27,779,405 pounds.

The production of refined oils during the period was as follows: Cottonseed 273,120,362 pounds; coconut, 63,838,821 pounds; peanut, 1,485,101 pounds; corn, 18,112,611 pounds; soya-bean, 647,962 pounds; and palm-kernel, 389,730 pounds. The quantity of crude oil used in the production of each of these refined oils is included in the figures of crude consumed.

The data for the factory production, factory consumption, imports, exports, and factory and warehouse stocks of fats and oils and for the raw materials used in the production of vegetable oils for the three-month period appear in the following statements:

PRODUCTION, CONSUMPTION AND STOCKS OF FATS AND OILS

(In some cases, where products were made by a continuous process, the intermediate products were not reported)

Kind	Factory operations for the quarter ended June 30, 1927		Factory and Warehouse stocks
	Production (pounds)	Consumption (pounds)	June 30, 1927 (pounds)
Vegetable Oils:[1]			
Cottonseed, crude	204,902,851	305,997,944	32,014,452
Cottonseed, refined	273,120,362	250,318,819	461,059,179
Peanut, virgin and crude	1,399,915	1,742,408	1,543,750
Peanut, refined	1,485,101	1,056,891	912,423
Coconut, or copra, crude	71,031,662	138,381,628	108,434,207
Coconut, or copra, refined	63,838,821	55,274,848	15,544,824
Corn, crude	27,779,405	24,446,264	13,150,995
Corn, refined	18,112,611	2,659,811	8,017,778
Soya-bean, crude	776,259	1,777,547	8,522,896
Soya-bean, refined	647,962	882,390	1,388,889
Olive, edible	.....	594,162	5,027,319
Olive, inedible	.....	2,004,010	1,063,255
Sulphur oil, or olive foots	.....	9,930,304	8,388,158
Palm-kernel, crude	.....	840,483	1,485,830
Palm-kernel, refined	389,730	39,436	212,845
Rapeseed	.....	3,647,768	4,966,972
Linseed	167,232,121	120,146,543	152,628,386
Chinese wood or tung	.....	20,524,997	20,967,938
Chinese vegetable tallow	.....	1,502,590	2,310,754
Castor	13,895,276	4,955,201	8,745,743
Palm	.....	23,159,446	22,329,071
All other	183,206	1,982,779	4,899,342

Kind	Factory operations for the quarter ended June 30, 1927 Production (pounds)	Consumption (pounds)	Factory and Warehouse stocks June 30, 1927 (pounds)
Fish Oils:[1]			
Cod and cod-liver	278,490	3,978,614	6,648,615
Menhaden	1,706,345	12,822,232	11,809,725
Whale	1,607,250	17,764,482	41,173,140
Herring, including sardine	3,143,198	6,993,589	9,261,650
Sperm	25,125	249,363	2,012,729
All other, including marine animal	248,025	1,027,874	1,662,962
Animal Fats:			
Lard, neutral	15,468,110	6,182,524	8,392,551
Lard, other edible	443,334,219	2,588,061	113,975,155
Tallow, edible	13,039,210	9,767,095	5,184,608
Tallow, inedible	109,139,195	150,747,424	94,645,361
Neat's-foot oil	2,352,441	1,837,039	1,755,047
Greases:			
White	21,762,628	8,642,498	8,357,441
Yellow	17,704,527	10,239,690	8,457,810
Brown	11,950,232	10,070,075	4,387,534
Bone	6,542,876	281,564	2,420,144
Tankage	13,373,548	268,188	3,598,098
Garbage or house	25,635,497	16,556,526	15,590,893
Wool	2,220,423	1,753,530	3,354,480
Recovered	814,611	574,280	1,144,000
All other	2,611,277	1,194,237	3,439,968
Other Products:			
Lard compounds and other lard substitutes	253,857,753	439,024	23,704,553
Hydrogenated oils	114,393,199	107,989,899	11,435,228
Stearin, vegetable	3,835,787	3,985,561	1,795,722
Stearin, animal, edible	17,514,098	12,150,184	8,561,114
Stearin, animal, inedible	5,712,610	7,125,462	4,048,847
Oleo oil	35,803,544	12,034,682	8,595,348
Lard oil	6,793,433	5,183,464	4,764,459
Tallow oil	3,081,617	2,001,862	1,763,106
Fatty acids	42,562,270	45,847,981	5,799,313
Fatty acids, distilled	8,742,804	10,100,784	4,040,356
Red oil	12,475,757	6,202,555	7,333,095
Stearic acid	7,987,555	2,365,437	3,875,063
Glycerin, crude 80% basis	32,975,380	32,003,769	13,468,757
Glycerin, dynamite	11,476,612	6,475,571	15,657,439
Glycerin, chemically pure	15,096,215	1,703,485	9,465,417
Cottonseed foots, 50% basis	63,974,171	77,648,154	52,844,735
Cottonseed foots, distilled	49,361,727	44,908,181	7,946,093
Other vegetable oil foots	13,832,324	11,339,709	920,154
Other vegetable oil foots, distilled	756,366	249,819	362,833
Acidulated soap stock	22,928,923	21,299,781	16,832,890
Miscellaneous soap stock	29,258	79,313	42,845

[1]The data of oils produced, consumed and on hand by fish oil producers and fish canners were collected by the Bureau of Fisheries.

Raw Materials Used in the Manufacture of Vegetable Oils

Kind	Tons of 2,000 pounds: Consumed March 31 to June 30	Tons of 2,000 pounds: On hand June 30
Cottonseed	672,437	101,391
Peanuts, hulled	2,063	598
Peanuts, in the hull	66	35
Copra	54,839	11,153
Coconuts and skins	1,432	98
Corn germs	52,606	538
Flaxseed	250,970	97,730
Castor beans	15,326	5,109
Mustard seed	191	1,382
Soya-beans	3,016	254
Other kinds	288	33

Imports of Oil Seeds, Quarter Ended June 30, 1927

Kind	Tons	Kind	Tons
Cottonseed	365	Poppy seed	405
Castor beans	19,268	Perilla and sesame seed	656
Copra	53,687	Other oil seeds	2,128
Flaxseed	214,511		

Imports of Foreign Fats and Oils, Quarter Ended June 30, 1927

Kind	Pounds	Kind	Pounds
Whale oil	37,481,752	Palm-kernel oil	4,175,009
Cod and cod-liver	6,911,310	Peanut oil	752,987
Other fish oils	3,654,465	Rape oil	5,254,462
Beef and hog fats	937,576	Linseed oil	388,652
Wool grease	3,695,649	Sesame oil	269,847
Grease and oils, n. e. s. (value)	$126,465	Soya-bean oil	6,482,103
Chinese wood oil or nut oil	36,137,664	Vegetable tallow	1,991,989
Coconut oil	82,959,165	Vegetable wax	2,774,457
Olive oil, edible	31,895,634	Other vegetable oils	2,331,574
Sulphur oil, or olive foots	12,266,880	Glycerin, crude	2,505,269
Olive oil, denatured	2,071,304	Glycerin, refined	2,464,362
Palm oil	31,756,562		

Exports of Foreign Fats and Oils, Quarter Ended June 30, 1927

Kind	Pounds	Kind	Pounds
Fish oils	7,903	Palm and palm-kernel oil	755,023
Other animal oils, fats and greases	5,600	Peanut oil	13,386
Chinese wood oil or nut oil	1,099,429	Soya-bean oil	761,756
Coconut oil	1,160,328	Other vegetable oils	186,881
Olive oil, edible	34,932	Vegetable wax	286,225

Exports of Domestic Fats and Oils, Quarter Ended June 30, 1927

Kind	Pounds	Kind	Pounds
Oleo oil	23,416,150	Other animal greases, oils and fats	21,718,132
Neat's-foot oil	614,151	Coconut oil	5,693,655
Other animal oils	113,966	Cottonseed oil, crude	15,541,946
Fish oils	142,450	Cottonseed oil, refined	4,287,978
Oleo stock	3,999,440	Linseed oil	599,953
Tallow	1,604,549	Soya-bean oil	1,114,869
Lard	198,167,352	Corn oil	30,757
Lard, neutral	6,582,588	Vegetable oil lard compound	1,389,520
Lard compound containing animal fats	1,878,838	Vegetable soap stock	1,930,722
Oleo and lard stearin	1,769,152	Other vegetable oils and fats	2,393,866
Grease stearin	1,034,396	Glycerin	103,852
Oleic acid, or red oil	395,356		
Stearic acid	577,414		